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TEMPORAL VARIABILITY MEASUREMENT OF SPECIFIC VOLATILE ORGANIC COMPOUNDS

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Methodology was developed to unambiguously determine trace levels of volatile organic compounds as they vary in concentration over a variety of time scales. This capability is important because volatile organic compounds (VOCs) are usually measured by time-integrative techniques that average out peak exposures to insignificance. The specific method presented here involves a preprogrammed sequential syringe sampler that can fill 150-cm^3 syringes with air at a rate of 2 to 90 min per syringe. The 12 collected samples are then transported to the laboratory for fully automated gas chromatographic separation using mass spectrometric detection. The instrumentation and method are described, and representative results are given to document the variability in volatile organic compound (VOC) concentrations in situations such as use of household products and water outgassing in residential air, automobile indoor air during driving, and office indoor air that is subject to ventilation system cycling. The method is shown to perform automatically in both sampling and analytical modes. Contamination and sample integrity tests show typical precision to be about 10% relative standard deviation. Field tests show that VOC concentrations can vary by greater than an order of magnitude on different time scales, ranging from 2 min to 24 h.

KEY WORDS: Indoor air, VOCs, automated, time-resolved, variability, organics.

INTRODUCTION

The identification and quantification of trace-level volatile organic compounds (VOCs) in the breathing space have become important because some of these compounds have been linked to adverse health effects in humans.[1–5] A variety of techniques are in use today for the determination of VOC concentrations, most of which rely on time-integrated sampling of air through solid adsorbents or on collection of whole-air samples in containers for subsequent laboratory analysis. In addition, semi-real-time techniques based on portable gas chromatographic (GC) instruments have been developed; however, these techniques are generally used only as screening tools because of the limited GC resolution and their reliance on

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nonspecific detectors. Though too numerous to list individually, many of the VOC determination techniques are discussed elsewhere.[6–8]

Studies based upon grab (10–20 s) samples[9] or time-integrated (5 to 24 h) samples[10,11] have shown that indoor air often contains VOC concentrations that are orders of magnitude higher than the corresponding outdoor air. This indicates the existence of local indoor sources, which generally contribute to the VOC burden on different use-dependent time frames.

For instance, VOCs released from stored paint or other volatile substances are generally emitted at low concentrations for long periods of time, whereas VOCs from cleaning products are emitted at high levels for very short periods. Because sampling for trace levels of VOCs is generally performed by using time-integrative techniques or by grab samples, as mentioned above, the short-term emissions may be either averaged to insignificance over the long sampling periods or totally missed by the very short sampling periods.

The previous development work in our laboratory has involved whole-air collection in 6-L SUMMA®-polished canisters (Scientific Instrumentation Specialists, Moscow, ID), reduced temperature preconcentration of VOCs from the whole-air sample, and subsequent analysis by high-resolution gas chromatography and mass selective detection (GC/MSD). This technique results in high sensitivities and unambiguous identification for specific compounds, but is generally restricted to individual grab samples or time-integrated samples because of the logistics of handling the canisters, both during sampling and analysis.[9,12–18] To investigate the time-dependent behavior of VOCs in indoor air without the problems associated with handling numerous individual canisters, yet still maintain the analytical quality and sensitivity of laboratory GC/MSD analysis, we have developed a monitoring procedure including an improved and fully automated sampling technique with *in situ* sequential collection of discrete volumes of whole air.

The sampling instrument is a commercially available unit that has been modified to our specifications. It is portable and can be programmed to fill 12 stainless-steel syringes (150 cm^3 each) consecutively at a rate of 2 to 90 min per sample. The GC/MSD method is fully automated to sequentially analyze the 12 syringe samples and generate reports of concentrations of 40 specific VOCs. The automated analysis aspects required modification of the sampling instrument and a specially designed sample transfer manifold and sampler-to-analyzer interface.

In this paper, the implementation of the automated syringe sampler collection and GC/MSD analysis method is discussed, and data from a number of representative experiments are presented to document the time-dependent variability of VOC concentrations and the usefulness of the new monitoring procedures.

EXPERIMENTAL SECTION

Equipment

The sequential syringe sampler was purchased from Demaray Scientific Instru-

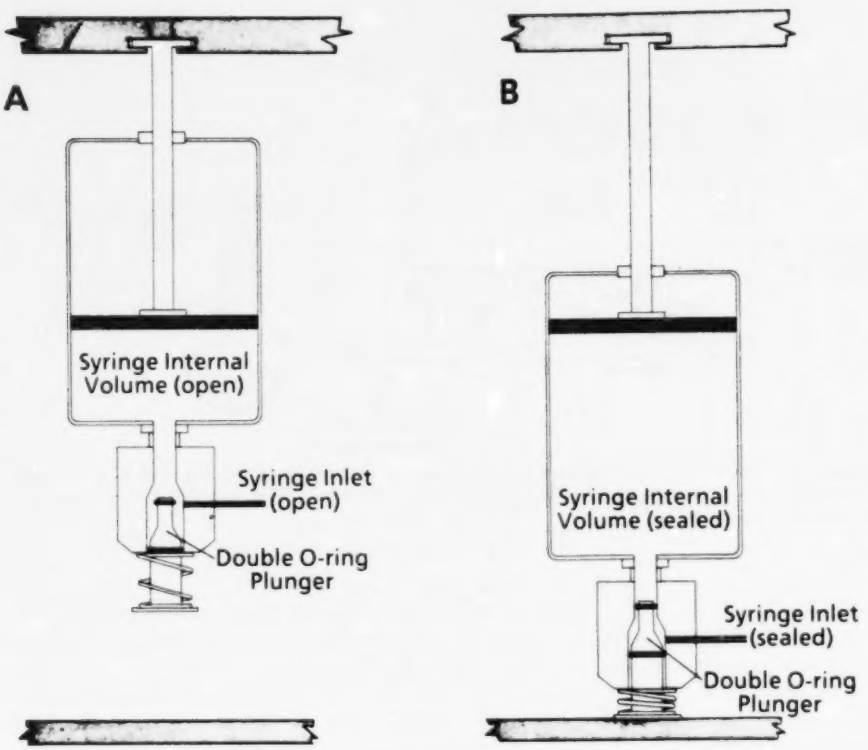

Figure 1 Syringe sealing assembly. (A) Plunger allows transfer of sample into or out of syringe. (B) Plunger seals syringe contents from inlet tube.

ments Ltd. (Pullman, WA), and modified to our specifications. The instrument consists of an array of 12 syringe drives that can accommodate syringes up to 150 cm^3 in volume. The syringes used for VOC work are internally SUMMA® polished (a proprietary technique that passivates the metal surfaces to minimize VOC adsorption) and include a double O-ring plunger as a sealing mechanism. This plunger assembly was modified to allow both closure of the sample volume at the end of sample collection and automatic release at the start of an analysis. A diagram of the modified assembly is given in Figures 1A and 1B. The syringe drives are programmable through an attached keypad that allows automatic sampling-sequence initiation based on a real-time clock; thus we can set the time-per-sample in a range of 2 to 90 min, and we can set the delay time between syringe samples as an integral multiple of the time-per-sample from 0 to 15. The sampler can operate off the power supplied by its internal battery system for at least 48 h. The electronics were modified with a two-position switch to select between normal sequential operation and operation triggered by an external signal. The "normal" position is used during sample collection, and the "external"

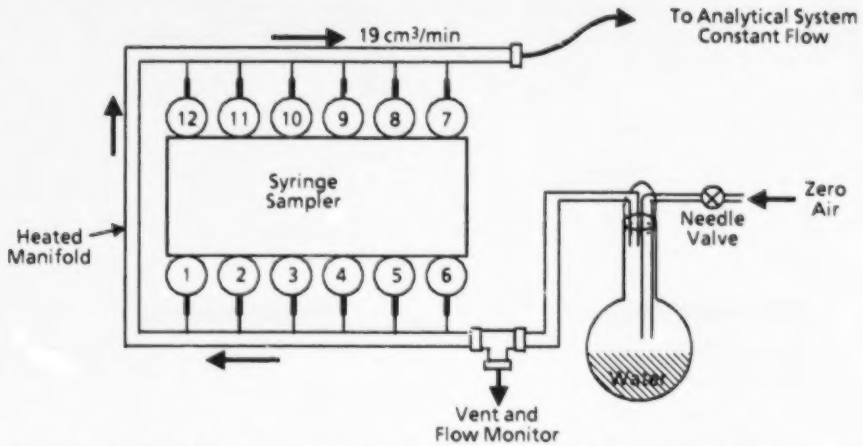

Figure 2 Manifold for transferring syringe sample to the analytical system. Contents of each individual syringe are injected into the manifold purge flow and swept into the analytical system cryogenic preconcentration trap.

position is used during sample analysis when the analytical system signals the appropriate time for sample transfer. A second switch was added that selects drive motor direction to either draw the sample into the syringes or reverse the flow to eject the sample from the syringes. Although Demaray no longer manufactures this instrument, it is now available from Scientific Instrumentation Specialists, Inc. (Moscow, ID), and our modifications are included as options.

To transfer samples to the analytical instrument, a continuously purged, heated manifold is attached to each syringe. The manifold is composed of high-grade nickel tubing (0.25-in o.d.) with 12 welded ports of 316-grade stainless-steel tubing (0.125-in o.d.) and is kept heated to 60 °C. Teflon® tubing is used to connect the ports to the moving syringes. Upon activation, an individual syringe injects its contents into the manifold's transfer gas, which is collected by the analytical system's cryogenic preconcentrator. Because the analytical system pulls samples at a rate of 19 cm^3/min, the syringe rate is set for 12.5 cm^3/min to displace only a portion of the purge flow. In this manner, the entire 150-cm^3 sample volume is collected as a slug entrained in the purge gas. Note that for a nominal compound of molecular weight 100 g/mole, a 1-ppbv concentration in a 150-cm^3 volume at 25 °C corresponds to about 0.6 ng of analyte. Experience has shown that water vapor tends to preferentially deactivate chemisorption sites on metal surfaces that would otherwise capture compounds of interest. Therefore, the transfer gas is passed through an in-line humidifier ($RH \simeq 90\%$) prior to entering the syringe manifold to facilitate analyte transfer. A diagram of this arrangement is given in Figure 2.

The analytical system consists of a modified model 320-01 cryogenic preconcentrator (Nutech Corp., Durham, NC) coupled to a model HP-5880 GC (Hewlett-

Packard, Avondale, PA). This arrangement is described in detail in other works.[14-16,18] For this study, the chromatographic detector used was a model HP-5970B MSD (Hewlett-Packard, Palo Alto, CA), equipped with a Hewlett-Packard series 200 HP-9816 computer. This GC/MSD system is programmed for automatic, repetitive analysis that includes data reduction and report generation. The system is set up to determine 40 compounds of environmental interest at the parts-per-billion-by-volume level, with a sensitivity of about 0.2 ppbv per compound in a 150-cm^3 whole-air sample. It has been used previously in conjunction with canister sampling in other EPA analysis projects.[12-19]

Sample Collection and Analysis Procedures

In the laboratory, before sample collection is begun, the syringes are flushed three times with heated zero-grade air while they are attached to the manifold. The time base, delay time, and automatic start feature are set, and the sampling mode switch is set to the "collect" position.

After sample collection, the system is returned to the laboratory, the syringes are connected to the manifold, and the interface cable is connected to the analytical system. A humidified, zero-air purge flow (which also acts as a sample transfer flow) is established in the manifold. The manifold temperature is kept at about 55 °C with a resistive heater. The analytical system draws a constant sample flow of 19 cm^3/min from the manifold compared to a syringe exhaust rate of 12.5 cm^3/min. The trapping cycle is set for a total of 24 min, twice as long as the syringe injection, to ensure that the syringe contents are swept through the manifold and into the trap.

Upon completion of sample trapping, the GC sampling valve is cycled to the inject position, and the cryotrap is heated. The trapped analytes are then injected onto a nonpolar capillary column for analysis. Detailed procedures and listings for the GC analytical programming, the data acquisition programming, and the data reduction macro programs are available.[20] The MSD computer is set up to analyze 14 samples in order: 1 for the purge gas, 12 for the syringe samples, and 1 more for the purge gas. The two purge gas samples serve as checks of the cleanliness of the manifold and transfer gas. Analytical procedures are repeated automatically after each analysis, resulting in a complete set of calibrated analyses with no operator intervention.

Laboratory Tests

Controlled laboratory experiments were performed to determine the expected background, precision, and approximate accuracy of the syringe sampler methodology. These included both contamination and sample integrity tests.

To test for syringe contamination, the sampler was set up for analysis in the laboratory. Syringes were flushed twice each with humidified and dry zero-air. A full analytical cycle was then initiated. During the first run, the syringes were filled

with the manifold transfer gas while this gas was being analyzed simultaneously. The contents of the 12 syringes was then analyzed, and an analysis was conducted on the manifold gas. The first and last analytical runs served as controls.

Sample integrity was determined with a controlled test sample prepared by filling a 6-L sample canister with ambient laboratory air and then spiking this matrix with 40 compounds of interest at the 0.6- to 4-ppbv level. This mixture is preferred over a synthetic mixture in zero-air because it simulates an actual ambient sample in that the various atmospheric constituents, such as H_2O vapor, CO_2, NO, and NO_2, are present as complicating factors. The sample was introduced into the manifold, and all 12 syringes were slowly filled (6 min per syringe) while the analytical system analyzed the sample twice. These two analyses served as controls. The sample gas was then removed, and the zero-air transfer flow was re-established in the manifold. After a 16-h (overnight) delay, a normal analytical sequence was initiated. As usual, the sequence included pre- and post-syringe analyses of the transfer gas so that trace contamination could be taken into account.

Field Experiments

A variety of field tests were conducted to demonstrate the utility of this technique and also to document the concentration variability of certain VOCs in indoor environments. The air matrices sampled include the following:

1) Residential bathroom air during morning shower activities;
2) Automobile interior air during driving on rural, urban, and highway roads;
3) Residential air during use of various household spray products;
4) Office air in a laboratory building over a 24-h period; and
5) Kitchen air during cooking activities using a gas stove.

In the bathroom air test, the possibility of VOCs outgassing from hot water from a shower was explored. Sample time was set for 2 min per syringe with a zero delay setting between samples; the bathroom door was kept closed only during the time the shower was on. Two "background" samples, five "shower on" samples, and five subsequent "dilution" samples were taken.

For the second field test, the sampler's ability to operate off its internal battery allowed sample collection of an automobile's interior air during a typical commute to work. The sampler was set to collect at 4 min per syringe with zero delays.

In the third field test, the impact of VOC releases from short-term use of household products was investigated to determine both peak exposures and dissipation rates when a very high level of VOC was released in a constricted space, a residential environment. This experiment was set up to measure exposures in a room adjoining the area of product use. The system was set up in the dining room of a private residence with a forced-air ventilation system, and five common spray products were used as VOC sources. These products were a stainless steel cleaner, a spot remover for fabrics, a dry film lubricant, a clear wood finish, and

an insect spray. The sampler was programmed to fill each syringe in 2 min with an 8-min delay between syringes to achieve time coverage of 2 h. After the first two syringes were filled (to serve as background samples), the household products were each sprayed for 15 sec onto cardboard near the air return of the ventilation system, at least 30 ft away from the sampler.

Two further tests of interest were performed, the first in an office area within our laboratory building and the second in a residential kitchen during meal preparation on a gas stove. The office test was performed to cover a 24-h period by sampling 30 min per syringe with a 90-min delay between syringes, whereas the kitchen test time scale was set at 4 min per syringe and zero delay to cover cooking time of 48 min. Use of natural gas as a heat source for cooking was investigated by altering the analytical procedure slightly. Rather than operating the mass spectrometer to specifically determine only the 40 compounds of environmental interest as in the preceding tests, we used the mass spectrometer in full-scan mode to allow detection of a variety of hydrocarbon species. This more general procedure, however, does sacrifice sensitivity and reduces certainty in the quantitation.

RESULTS AND DISCUSSION

Tests using zero-air as a synthetic sample showed that the syringe sampler technique is essentially contamination-free. Background levels were typically less than 0.05 ppbv. The only exceptions are encountered when a previous sample set contains a very high level of a particular compound, which then can show some residual amounts in a subsequent test, typically 2 orders of magnitude below the previous sample's concentration. This carryover is probably caused by absorption in the deformable materials used in the slip-seals of the syringe, a problem solved with additional flushing prior to sampling; typically, one flushing cycle reduced carryover concentrations by an order of magnitude.

The sample integrity tests showed syringe-to-syringe variability of typically 10% (range 1.5 to ~25%) relative standard deviation (RSD) in the concentration range of 0.6 to about 4 ppbv. For some compounds, a trend of decreasing concentration with increasing syringe number was noted. Because each analysis takes more than an hour to complete, this could indicate some sample loss due to the increased storage time for the samples. This is not an overwhelming problem, however, because the overall trend (when observed) generally is not more than about a 10% change in sample concentration. In general, these controlled tests have shown that the method is useful when trends and approximate quantitation (±10%) are required. Specific details of these tests on a per-compound basis are available.[20]

The purpose of the field tests of the syringe sampling and analysis method was to demonstrate its general utility and applicability to actual sampling studies by documenting the VOC variability in some representative situations. The tests performed showed that indoor VOC concentrations for specific compounds can, depending upon the setting, vary greatly over short periods of time. Though a variety of tests were performed, each resulting in time-dependent data for 40

compounds of specific environmental interest, only selected highlights are discussed.

In the bathroom air test, the only one of the compounds monitored that demonstrated a strong correlation to the water source was trichloromethane, which had a background level that ranged from ~3 ppbv to a maximum 97 ppbv just before the end of the shower. Certain other VOCs showed a definite decreasing trend throughout the test, regardless of the shower status, indicating a previous release that was being diluted slowly. The compounds showing this trend included various aromatic hydrocarbons, such as ethylbenzene, xylene isomers, and trimethylbenzene isomers. Of particular concern was the surprisingly high level of *p*-dichlorobenzene that slowly decreased from about 50 to 30 ppbv during the test. The hydrocarbons were most likely the result of the prior use of a hairspray product, and the source of the dichlorobenzene was possibly an air freshener spray. Other detected compounds, particularly the halocarbons Freon 11, 12, and 114, tri- and tetrachloroethene, etc., showed constant background levels in the low-part-per-billion-by-volume range. The time-dependent concentrations of trichloromethane and *p*-dichlorobenzene during the shower test are plotted in Figure 3.

In the second field test during periods of high traffic and low-speed driving, the compounds associated with gasoline vapors and combustion products increased by as much as a factor of 4 or 5 over the rural driving value. These compounds included the aromatic hydrocarbons, such as benzene, toluene, xylenes, styrene, and trimethylbenzenes. Typical concentrations ranged from 1 to 20 ppbv. The concentrations of the halocarbons Freon 11, Freon 12, and dichloromethane tended to increase when the warm car was stationary, indicating a local source. Only one of the monitored compounds, carbon tetrachloride, demonstrated an essentially stable background level regardless of driving conditions. The expected value of ~0.1 ppbv (the global background concentration in the absence of a local source) indicates that the equipment was operating properly. The remaining 28 compounds (halogenated hydrocarbons) were not detected. The time dependence of representative aromatics is presented in Figure 4.

As expected in the third field test, in which five spray products were used as VOC sources in a residence, the major components of these products that overlapped with our VOC list exhibited an initial high concentration upon release (e.g., *m,p*-xylene, 1,1,1-trichloroethane, and dichloromethane), as shown in Figure 5(A). However, the dilution due to air exchange did not occur uniformly for these compounds. The concentration of dichloromethane indicates a secondary release, and the *m,p*-xylene concentration profile shows residual, low-level outgassing. For the very low level contaminants, Figure 5(B) shows that chloromethane was apparently not a constituent in the test products and that both trichloroethene and carbon tetrachloride were minor constituents of one or more of the products. The presence of carbon tetrachloride was unexpected because this chemical has been banned in consumer products.

The office test showed variability patterns dependent upon compound type. Two patterns of concentration were found that were attributable to the ventilation system, which was turned on at 6 a.m. and turned off at 6 p.m. during this test. The first showed an increase in chlorinated species such as Freon 12, Freon 113,

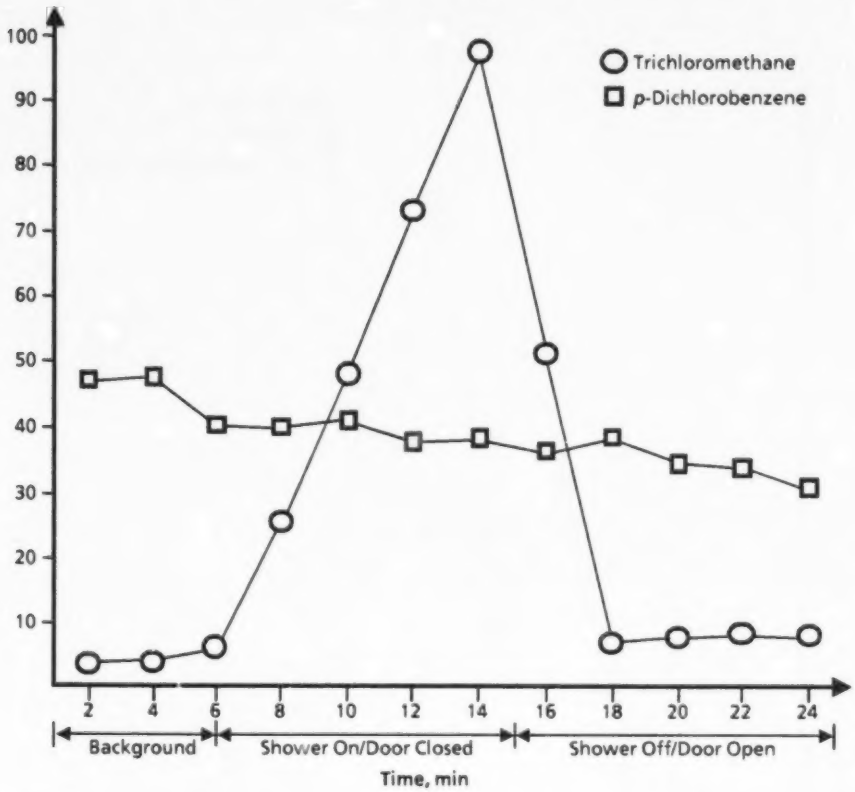

Figure 3 Bathroom air test during shower activity. A strong correlation is shown between the trichloromethane concentration and the length of shower (total time = 24 min). The *p*-dichlorobenzene shows a decreasing trend unrelated to shower activity, which indicates decay from an earlier emission.

trichloromethane, and 1,1,1-trichloroethane durng the time that the ventilation was off, which indicates that local sources of these compounds build up when the external exchange air is off. The second pattern involved aromatic hydrocarbons such as benzene, toluene, and xylenes, the levels of which increased in the morning when the air-handling system was first turned on. This may be caused by overnight outgassing of fuels in the first-floor portion of the building where automobile emissions research is performed; during the initial start-up of the ventilation system, these vapors could be distributed to the other areas of the building. Graphs showing examples of these patterns are presented in Figures 6(A) and (B). In addition, spurious unrelated changes in Freon 11 and dichloromethane concentrations were documented; however, these were not correlated to ventilation parameters.

Surprisingly, the kitchen test showed very little cooking-related time dependence

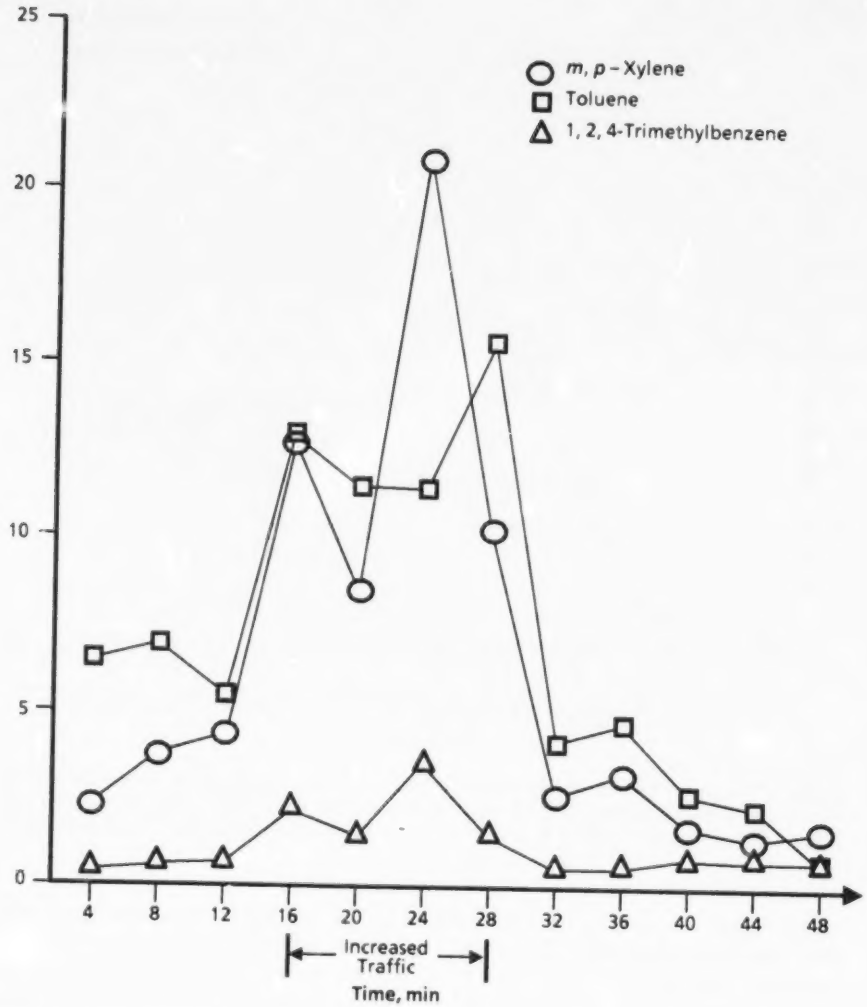

Figure 4 Automobile interior test during drive to work. Aromatic hydrocarbon concentrations are related to external traffic.

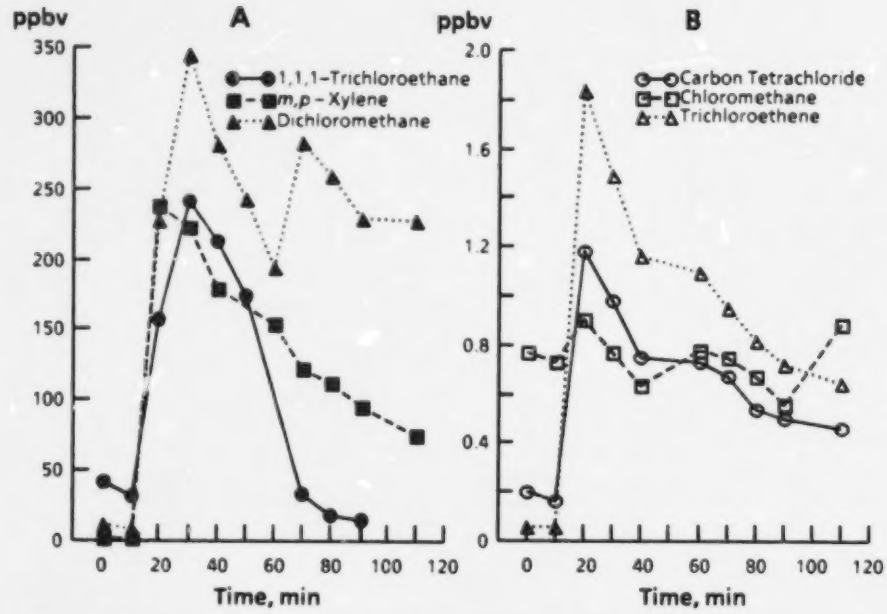

Figure 5 Short-term use of selected household products. (A) Some major constituents exhibit extremely high concentrations in a centrally located area, 30 ft from the source. (B) Some low-level concentrations indicate unexpected contaminants such as carbon tetrachloride. Chloromethane is not an apparent constituent of these products.

of any compound concentrations, but overall levels of alkane hydrocarbons, and also benzene and toluene, were slightly higher than those generally encountered in other residential settings.

CONCLUSIONS

The presented methodology for determining temporal variability of VOC concentrations in indoor air was shown to perform automatically in both the sampling and the analytical modes after modification of commercial hardware and through appropriate interfacing of commercially available sampling and analysis equipment. Laboratory tests showed that contamination levels for target compounds were well below typical sample levels and that sample integrity of a known mixture of analytes in an ambient air matrix could be preserved with 10% RSD for most compounds, errors that are generally considered negligible at trace levels. Though the system was not evaluated for the full range of possible combinations of temperature, relative humidity, and co-contaminants, the test data were taken under "typical" indoor air conditions using the laboratory ambient air as a matrix.

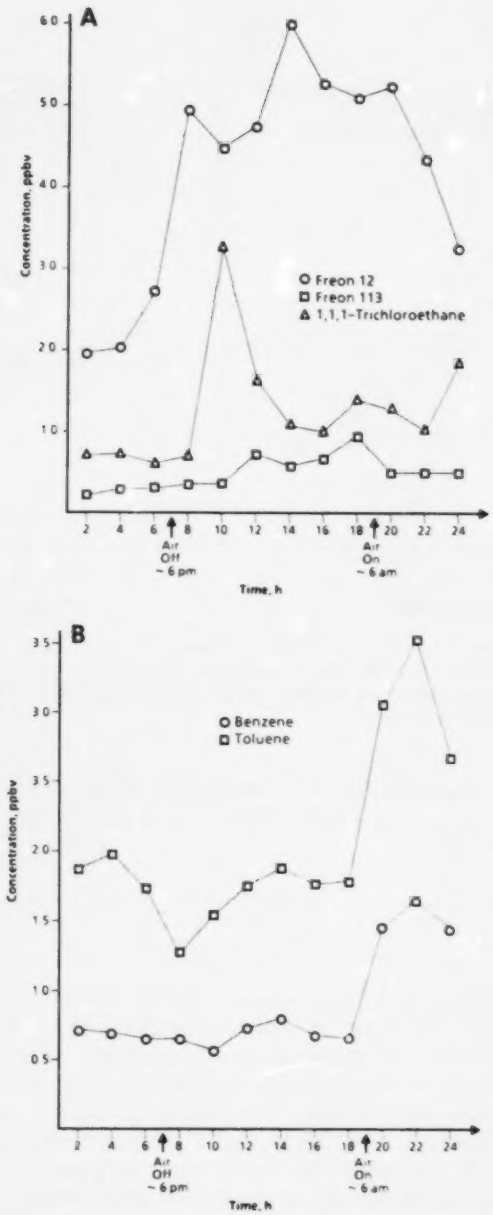

Figure 6 Office/laboratory building air test. Multiple patterns depending upon VOC species were found. (A) Halocarbons increase when ventilation rates are low. (B) Aromatic compounds increase when ventilation is first turned on.

As such, the test results are indicative of system performance under field conditions. Field tests in various situations showed that VOC concentrations can vary by over an order of magnitude on different time scales and that variability can be compound-dependent.

These results demonstrate the primary utility of the method, the unambiguous determination of peak exposure levels of VOCs. For instance, the chloroform exposure documented in the bathroom test (Figure 3) shows a short-term concentration increase up to about 100 ppbv (an average of about 50 ppbv over the 12-min-long shower) with a background level of about 2 ppbv. A 24-h integrated sample would yield only the time-weighted average of the chloroform exposure:

$$\frac{(12\ \text{min} * 50\ \text{ppbv}) + (1428\ \text{min} * 2\ \text{ppbv})}{1440\ \text{min}} = 2.4\ \text{ppbv}.$$

The epidemiological implications may be quite different for a 100-ppbv peak exposure than for a 2.4-ppbv average exposure.

We feel that the method is the appropriate choice when time-resolved VOC concentrations are required. The technique requires little effort or knowledge on the field operator's part because the sampler can be programmed for automated operation while in the laboratory and then transported to the sampling site. Battery operation obviates the need for external electric power, thus allowing sampling in cars and airplanes. Finally, the instrument modifications and techniques developed to allow fully automated analysis of the samples greatly reduce the analytical effort and cost that would be expended using existing methodology.

Disclaimer

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